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CITATION:

OKAMURA, Keizo ...[et al]. <Preliminary>Change of X-ray Diffraction Peaks in Aliphatic Cellulose Ester Homologues. Wood research : bulletin of the Wood Research Institute Kyoto University 1983, 69: 89-90

ISSUE DATE:

1983-03-25

URL:

<http://hdl.handle.net/2433/53331>

RIGHT:

Change of X-ray Diffraction Peaks in Aliphatic Cellulose Ester Homologues

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Abstract—While characterizing aliphatic cellulose ester homologues using various instruments we have noticed that their first (the largest) X-ray diffraction peak always moved toward smaller angle (larger spacing) as the number of carbon in the acyl group was increased. This phenomenon might give us an insight of the molecular structure of the cellulose ester homologues, and it is reported as a preliminary.

Experimental

A Whatman cellulose powder CF-11 was used as a starting cellulose sample. Before the esterification of cellulose an esterification mixture was prepared: three moles of trifluoroacetic anhydride and three moles of saturated normal acid were mixed, and kept at 50°C for 20 min with continuous stirring. One mole of the cellulose sample was added to the solution in Erlenmeyer flask equipped with a condenser, and esterified at 50°C for 5 hr with stirring. At the end of esterification reaction, the solution was poured into 20 volume of methyl alcohol, and the resultant precipitate was filtered, washed 5 times with ethyl alcohol, and resuspended in a large excess of methyl alcohol for seven days. The precipitate was again filtered, washed with methyl alcohol, ethyl ether and finally with hot ethyl alcohol before vacuum drying. The samples prepared were per-acylated and their weight average molecular weights were the order of 10^5 .

About 200 mg of ester sample was pressed into a disk in a KBr disk mold. Nickel filtered $\text{CuK}\alpha$ X-rays generated at 35 KV, 20 mA were employed, and the intensities were recorded on a strip-chart with Rigaku standard type Geigerflex 2011B diffractometer.

Results and Discussion

Figure 1 is the X-ray diffractograms of the original Whatman cellulose and

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ester homologues therefrom. As the number of carbon in acyl group increases from three up to twelve it is noticed that the first (the largest) diffraction peak moves toward smaller angle (larger interplanar spacing). This is clearly understood when the interplanar spacings were plotted against the number of carbon in acyl group of cellulose esters (Figure 2); the interplanar spacing of cellulose propionate was 1.17 nm while it reached about 2.99 nm in the case of cellulose laurate.

The significance of the shift of the first diffraction peak is not known at the present time, but the authors presume that the values must somehow be related to the distance between the neighboring two cellulose chains in unit lattice, which increases as the length of acyl group becomes longer.

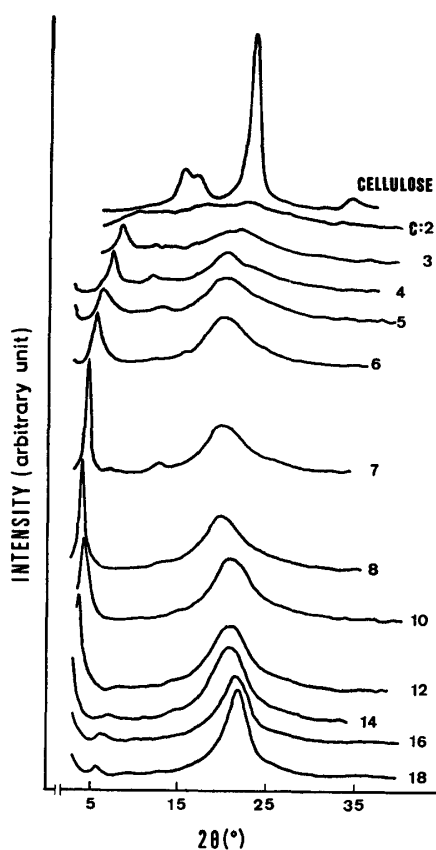


Fig. 1. X-ray diffractograms of cellulose and cellulose esters. C: number of carbon in acyl group.

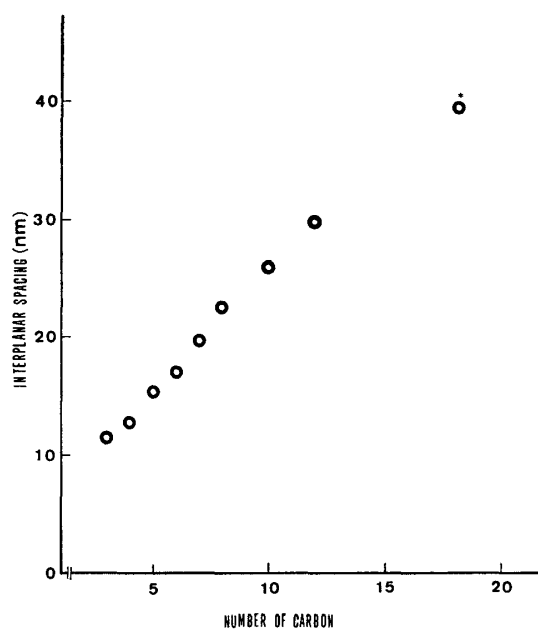


Fig. 2. Interplanar spacing vs number of carbon in acyl group of cellulose esters.
*: obtained from small angle diffraction.